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प्लास्टिक — परीक्षण पद्धतियाँ

भाग 11 विशेष गुणधर्म

अनुभाग 9 कैपिलरी विस्कोमीटर के उपयोग से तनु विलयन में पॉलिमर की श्यानता ज्ञात करना — साधारण सिद्धांत

Indian Standard

PLASTICS — METHODS OF TESTING

PART 11 SPECIAL PROPERTIES

Section 9 Determination of the Viscosity of Polymers in Dilute Solution Using Capillary Viscometers — General Principles

ICS 83.080.01

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

December 2004 **Price Group 8**

Plastics Sectional Committee, PCD 12

NATIONAL FOREWORD

This Indian Standard (Part 11/Sec 9) which is identical with ISO 1628-1 : 1998 'Plastics — Determination of the viscosity of polymers in dilute solution using capillary viscometers — Part 1 : General principles' issued by the International Organization for Standardization (ISO) was adopted by the Bureau of Indian Standards on the recommendations of the Plastics Sectional Committee and approval of the Petroleum, Coal and Related Products Division Council.

The text of ISO Standard has been proposed to be approved as suitable for publication as an Indian Standard without deviations. Certain conventions are, however, not identical to those used in Indian Standards. Attention is particularly drawn to the following:

a) Wherever the words 'International Standard' appear referring to this standard, they should be read as 'Indian Standard'.

b) Comma (,) has been used as a decimal marker while in Indian Standards, the current practice is to use a point (.) as the decimal marker.

CROSS REFERENCES

In this adopted standard reference appears to certain International Standards listed below for which Indian Standards also exist. The corresponding Indian Standards, which are to be substituted in their place, are given below along with their degree of equivalence for the editions indicated. However, that International Standard cross-referred in this adopted ISO Standard which has subsequently been revised, position in respect of latest ISO Standard has been given:

International Standard	*Corresponding Indian Standard*	*Degree of Equivalence*
ISO 31-0 : 1992 Quantities and units — Part 0 : General principles	IS 1890 (Part 0) : 1995 Quantities and units: Part 0 General principles (*first revision*)	Identical
ISO 31-3 : 1992 Quantities and units — Part 3 : Mechanics	IS 1890 (Part 3) : 1995 Quantities and units: Part 3 Mechanics (*second revision*)	do
ISO 3105 : 1994 Glass capillary kinematic viscometers — Specifications and operating instructions	Nil	—
ISO 3205 : 1976 Preferred test temperatures	Nil	—

In case of ISO 3105 and ISO 3205, the Technical Committee responsible for the preparation of this standard took cognizance of these Standards and decided that they are acceptable for use in conjunction with this standard.

For tropical countries like India, the standard temperature and the relative humidity shall be taken as 27 ± 2°C and 65 ± 5 percent respectively.

In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

PLASTICS — METHODS OF TESTING

PART 11 SPECIAL PROPERTIES

Section 9 Determination of the Viscosity of Polymers in Dilute Solution Using Capillary Viscometers — General Principles

1 Scope

This part of ISO 1628 defines the general conditions for the determination of the reduced viscosity, intrinsic viscosity and K-value of organic polymers in dilute solution. It defines the standard parameters that are applied to viscosity measurement, and can be used to develop standards for measuring the viscosities in solution of individual types of polymer. It can also be used to measure and report the viscosities of polymers in solution for which no separate standards exist.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this part of ISO 1628. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this part of ISO 1628 are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 31-0:1992, *Quantities and units — Part 0: General principles.*

ISO 31-3:1992, *Quantities and units — Part 3: Mechanics.*

ISO 3105:1994, *Glass capillary kinematic viscometers — Specifications and operating instructions.*

ISO 3205:1976, *Preferred test temperatures.*

3 Definitions

3.1 Dimensions and units

The dimensions of properties defined in this part of ISO 1628 are expressed in terms of L for length, M for mass and T for time in accordance with ISO 31-0, while the units appropriate to the properties are given in ISO 31-0 and ISO 31-3.

3.2 Definitions applicable to any liquid

3.2.1 viscosity: The viscosity of a fluid sheared between two parallel plates, one of which moves relative to the other in uniform rectilinear motion in its own plane, is defined by the Newton equation

$$\tau = \eta \dot{\gamma} \qquad \text{...(1)}$$

where

τ is the shear stress;

η is the viscosity;

γ is the velocity gradient or rate of shear, given by $\frac{\mathrm{d}v}{\mathrm{d}z}$ where v is the velocity of one plane relative to the other and z the coordinate perpendicular to the two planes.

The dimensions of viscosity are: $\mathrm{ML^{-1}T^{-1}}$.

The units of viscosity are: Pa·s.

For practical use, the sub-multiple 10^{-3} Pa·s is more convenient.

NOTE — Viscosity is usually taken to mean "Newtonian viscosity", in which case the ratio of shearing stress to velocity gradient is constant. In non-Newtonian behaviour, which is the usual case with high-polymer solutions, the ratio varies with the shear rate. Such ratios are often called "apparent viscosities" at the corresponding shear rate.

3.2.2 viscosity/density ratio; kinematic viscosity, v: This ratio is defined by the equation

$$v = \frac{\eta}{\rho} \qquad \text{...(2)}$$

where ρ is the density of the fluid at the temperature at which the viscosity is measured.

The dimensions of kinematic viscosity are: $\mathrm{L^2T^{-1}}$.

The units of kinematic viscosity are $\mathrm{m^2 \cdot s^{-1}}$.

For practical use, the sub-multiple 10^{-6} $\mathrm{m^2 \cdot s^{-1}}$, i.e. $\mathrm{mm^2 \cdot s^{-1}}$, is more convenient.

3.3 Definitions applicable to polymer solutions

3.3.1 relative viscosity, η_r (also known as viscosity ratio): The ratio of the viscosity of the polymer solution (of stated concentration) η and the viscosity of the pure solvent η_0, at the same temperature:

$$\eta_r = \frac{\eta}{\eta_0} \qquad \text{...(3)}$$

The ratio has no dimensions.

3.3.2 relative viscosity increment (also known as viscosity ratio increment and specific viscosity): The viscosity ratio minus one:

$$\left(\frac{\eta}{\eta_0}\right) - 1 = \frac{\eta - \eta_0}{\eta_0} \qquad \text{...(4)}$$

The increment has no dimensions.

3.3.3 reduced viscosity, I (also known as viscosity number): The ratio of the viscosity ratio increment to the polymer concentration c in the solution:

$$I = \frac{\eta - \eta_0}{\eta_0\, c} \qquad \text{...(5)}$$

The dimensions of reduced viscosity are: L^3M^{-1}.

The units of reduced viscosity are m^3/kg.

For practical use, the sub-multiple 10^{-3} m^3/kg, i.e. cm^3/g, is more convenient and the commonly quoted numerical values for reduced viscosity (viscosity number) use these practical units.

The reduced viscosity is usually determined at low concentration (less than 5 kg/m^3, i.e. 0,005 g/cm^3), except in the case of polymers of low molar mass, for which higher concentrations may be necessary.

3.3.4 inherent viscosity (also known as logarithmic viscosity number): The ratio of the natural logarithm of the viscosity ratio to the polymer concentration in the solution:

$$\frac{\ln\left(\frac{\eta}{\eta_0}\right)}{c} \qquad \text{...(6)}$$

The dimensions and units are the same as those given in 3.3.3.

The inherent viscosity is usually determined at low concentration (less than 5 kg/m^3, i.e. 0,005 g/cm^3), except in the case of polymers of low molar mass, for which higher concentrations may be necessary.

3.3.5 intrinsic viscosity, $[\eta]$ (also known as limiting viscosity number): The limiting value of the reduced viscosity or of the inherent viscosity at infinite dilution:

$$[\eta] = \lim_{c \to 0} \left(\frac{\eta - \eta_0}{\eta_0\, c}\right)$$

$$\text{...(7)}$$

$$[\eta] = \lim_{c \to 0} \frac{\ln\left(\frac{\eta}{\eta_0}\right)}{c}$$

The dimensions and units are the same as those given in 3.3.3.

NOTE — The effect of the shear rate on the functions defined in 3.3.1 to 3.3.5 has been neglected, since this effect is usually negligible for values of the reduced viscosity, inherent viscosity and intrinsic viscosity less than 0,5 m^3/kg, i.e. 500 cm^3/g. Strictly speaking, all these functions should be defined at the limiting (preferably infinitely small) value of the shear rate.

3.3.6 ***K*****-value:** A constant independent of the concentration of the polymer solution and peculiar to the polymer sample. It is a measure of the average degree of polymerization.

$$K\text{-value} = 1\,000\,k \qquad \text{...(8)}$$

According to H. Fikentscher[2], k is calculated as follows:

$$\lg \eta_r = \left(\frac{75\,k^2}{1+150\,kc} + k \right) 100\,c$$

and therefore

$$k = \frac{1{,}5 \lg \eta_r - 1 + \sqrt{1 + \left(\frac{2}{c} + 2 + 1{,}5 \lg \eta_r \right) 1{,}5 \lg \eta_r}}{150 + 300\,c} \qquad \text{...(9)}$$

where

$\eta_r = \frac{\eta}{\eta_0}$ = the viscosity ratio (see 3.3.1);

c is the concentration, in 10^3 kg/m^3, i.e. g/cm^3.

A limiting viscosity number $[\eta]_k$ can be calculated from k:

$$[\eta]_k = 230{,}3\left(75\,k^2 + k\right)$$

4 Principle

The data needed for the evaluation of the functions defined in 3.3 are obtained by means of a capillary-tube viscometer. The efflux times of a given volume of solvent t_0 and of solution t are measured at fixed temperature and atmospheric-pressure conditions in the same viscometer. The efflux time of a liquid is related to its viscosity by the Poiseuille-Hagenbach-Couette equation:

$$v = \frac{\eta}{\rho} = kt - \left(\frac{A}{t^2} \right) \qquad \text{...(10)}$$

where

v is the viscosity/density ratio (see 3.2.2);

k is a constant of the viscometer;

A is a parameter of the kinetic-energy correction;

ρ is the density of the liquid;

t is the efflux time.

For the purposes of this part of ISO 1628, the kinetic energy correction $\left(\frac{A}{t^2}\right)$ shall be regarded as negligible when it is less than 3 % of the viscosity of the solvent. Hence equation (10) can be reduced to

$$v = \frac{\eta}{\rho} = kt \qquad \text{...(11)}$$

Moreover, if the solution concentrations are limited so that the solvent density ρ_0 and that of the solution ρ differ by less than 0,5 %, the viscosity ratio $\frac{\eta}{\eta_0}$ will be given by the so-called "efflux time ratio" $\frac{t}{t_0}$.

The need for these constraints, and the consequences of not observing them, is developed in annex B.

5 Apparatus

5.1 Capillary viscometer, suspended-level Ubbelohde type.

The use of a viscometer having the dimensions given in figure 1 or 2 is strongly recommended. Furthermore, it is strongly recommended that the size of the viscometer is chosen from among those listed in table 1. The choice is determined by the viscosity/density ratio of the solvent at the temperature of the measurement, as indicated in table 1. The next-smaller viscometer can also be used.

Other types of viscometer listed in ISO 3105 can be used, provided they give results equivalent to those given by the particular size of Ubbelohde viscometer chosen on the basis of the criteria specified in the preceding paragraph. In cases of dispute, an Ubbelohde viscometer shall be used.

With automated apparatus, fitted with special timing devices, it may be possible to obtain equivalent results with larger sizes of capillary than those listed for the appropriate solvent viscosity/density ratio in table 1.

5.2 Viscometer holder, suitable to hold the viscometer firmly in the thermostatic bath (5.3) in the vertical position.

5.3 Thermostatic bath, holding a transparent liquid or vapour and of such depth that, during the measurement, no portion of the test liquid will be less than 20 mm below the surface of the bath medium or less than 20 mm above the bottom of the bath.

The temperature control shall be such that, within the range 25 °C to 100 °C, the temperature of the bath does not vary from the specified temperature by more than 0,05 °C over the length of the viscometer, or between the viscometers if several determinations are carried out simultaneously.

At temperatures higher than 100 °C, the tolerance shall be ± 0,2 °C.

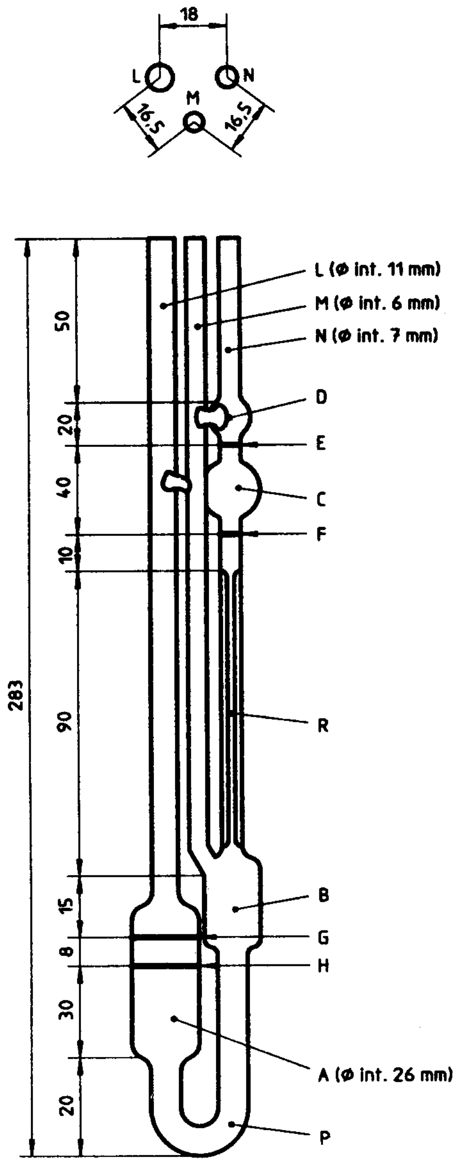
5.4 Temperature-measuring device.

A liquid-in-glass, "total immersion" thermometer, reading to 0,05 °C in the range to be used and in a known state of calibration, is suitable. Other thermometric devices of at least equal precision may be used.

5.5 Timing device.

Any timing device may be used providing that it can be read to 0,1 s and that its speed is constant to 0,1 % over 15 min.

Dimensions in millimetres

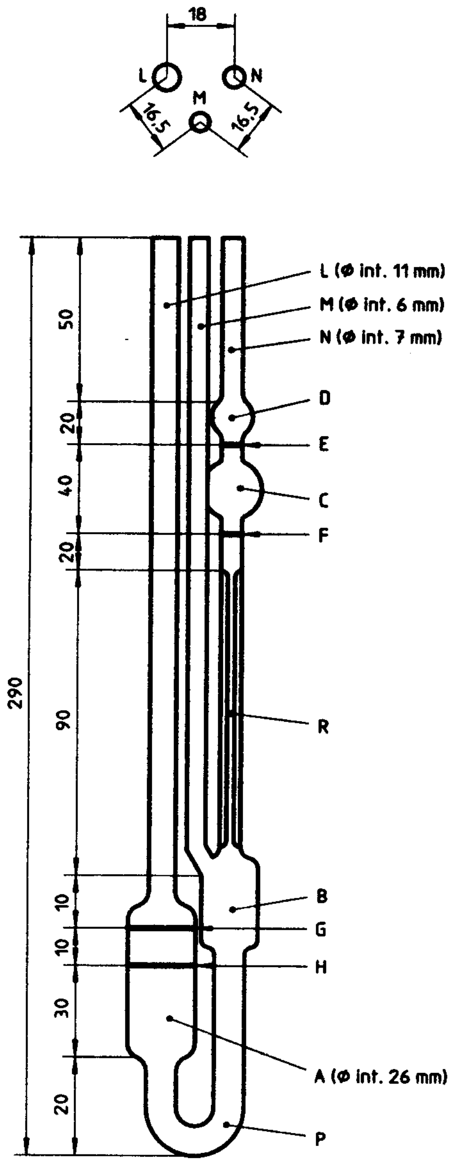


Graduation marks: E and F

Filling marks: G and H

Figure 1 — Ubbelohde viscometer

Dimensions in millimetres



Graduation marks: E and F

Filling marks: G and H

Figure 2 — DIN Ubbelohde viscometer

Table 1 — Ubbelohde viscometers recommended for the determination of the dilute-solution viscosity of polymers

Viscosity/density ratio of solvent at temperature of measurement	Ubbelohde conforming to ISO 3105:1994, table B.4		DIN Ubbelohde conforming to ISO 3105:1994, table B.9	
mm²·s⁻¹	Size No.	Diameter of capillary mm (± 2 %)	Size No.	Diameter of capillary mm (± 2 %)
0,15 to 0,30	0	0,24	0	0,36
0,31 to 0,50	0C	0,36	0c	0,47
0,51 to 0,75	0B	0,46	0a	0,53
0,76 to 1,50	1	0,58	I	0,63
1,51 to 2,50	1C	0,77	Ic	0,84
2,51 to 5,00	1B	0,88	Ia	0,95
5,01 to 15,00	2	1,03	II	1,13

6 Solutions

6.1 Preparation

The dissolution of the test sample of polymer in the solvent shall give a "true" solution, essentially free of microgels and associated macromolecules. Polymer degradation shall also be minimized. For these reasons, it is necessary for the dissolution procedure to be exactly defined and it is recommended that the following factors be specified:

a) the solvent and its pretreatment, if any;

b) the apparatus and the method of agitation;

c) the temperature range within which the system is maintained during the preparation of the solution;

d) the time interval necessary for the complete dissolution of the polymer without degradation, or at constant degradation;

e) the stabilizer and/or the protective atmosphere used;

f) the conditions of filtration of the solution, if applicable.

6.2 Concentration

Where no standard exists, careful consideration shall be given to the choice of solvent and the solution concentration. The solution concentration shall be chosen so that the ratio of the efflux time of the solution to the efflux time of the solvent is at least 1,2 and less than 2,0.

NOTE — A lower limit of 1,2 is necessary to ensure sufficient precision of the measured difference in efflux times. The upper limit of 2,0 is necessary because, at higher molecular masses, there can be shear effects and non-linearity of the viscosity number in relation to concentration.

More than one concentration can therefore be used for a given polymer/solvent system, depending on the molecular mass of the polymer under test.

The concentration is preferably expressed in kg/m^3 of solution or as the multiple 10^3 kg/m^3, i.e. g/cm^3.

7 Temperature of measurement

The temperature shall be chosen with due regard to sufficient solubility and other technical requirements, but kept constant for any particular polymer/solvent system. The temperature tolerance shall be specified. A temperature of 25 °C ± 0,05 °C shall be chosen whenever possible. If another temperature is used, it shall be chosen from the values recommended in ISO 3205 and stated in the test report.

8 Procedure

Measure the efflux times for the solution and the solvent successively in the same viscometer, using the following procedure:

8.1 Preparing and charging the viscometer

Maintain the bath at the specified test temperature.

Charge the dry, clean viscometer (for the cleaning procedure, see annex A) by tilting it about 30° from the vertical and pouring sufficient liquid through tube L (see figure 1 or 2) so that, when the viscometer is returned to the vertical, the meniscus is between the filling marks G and H. Avoid trapping air bubbles in the viscometer. The initial filling may be carried out away from the bath.

Mount the viscometer in a holder in the bath, ensuring that tube N is vertical. Allow time for the charged viscometer to reach the temperature of the bath. Usually 15 min will suffice if the measurement is to be made at 25 °C. At higher temperatures, longer times may be necessary. Unnecessary delays should be avoided as it is found that the most consistent results are obtained shortly after temperature equilibrium is attained with a freshly charged viscometer.

This procedure shall also be followed when a measured amount of solvent is added to a solution, already contained in the viscometer, in order to create a more dilute solution for additional determinations. The additional solvent shall be maintained at the specified test temperature prior to use.

In automated equipment, the viscometer is fixed in the vertical position within a temperature-controlled bath and the apparatus is designed to fill the viscometer with liquid in this position. The bath shall be maintained at the specified test temperature and an equilibration time selected in accordance with paragraph 3 of this subclause.

8.2 Efflux time measurement (refer to figure 1 or 2)

Close tube M and apply suction to tube N, or pressure to tube L, until the liquid reaches a level about 5 mm above mark E. Hold the liquid at this level by closing tube N.

Open tube M so that the liquid drops away from the lower end of capillary tube R.

When the liquid is clear of the end of the capillary and the lower end of tube M, open tube N. Measure the efflux time to the nearest 0,2 s as the time taken for the bottom of the meniscus to pass from the top edge of mark E to the top edge of mark F. For polymer solutions containing small amounts of finely divided pigments, e.g. carbon black, it may be necessary to view the top of the meniscus. Where large concentrations of pigments are involved, it may be necessary to centrifuge the solution before proceeding.

The opening and closing of the tubes may be carried out conveniently by means of taps or clips on clean flexible tubing attached to the ends of the tubes, making sure that no fluff or other contaminant enters the tube.

The first flow shall not be taken into account for measuring the efflux time. Repeat the measurement of efflux time immediately, without emptying or recharging, until two successive efflux times agree to within 0,25 %. Take the mean of these two values as the efflux time.

If two successive determinations of the mean efflux time of the solvent differ by more than 0,4 s, clean the viscometer (see annex A).

The efflux time for any "new" solution, created by the addition of solvent to an existing solution held in the viscometer, shall be determined as a separate liquid, in accordance with this procedure.

9 Expression of results

9.1 Reduced viscosity and intrinsic viscosity

Results are preferably expressed as a reduced viscosity.

The reduced viscosity I, in the units 10^{-3} m^3/kg, i.e. cm^3/g, is calculated from the equation

$$I=\frac{t-t_0}{t_0 c} \qquad \text{...(12)}$$

where

t is the efflux time of the solution, in seconds;

t_0 is the efflux time of the solvent, in seconds, through the same viscometer;

c is the concentration, expressed as 10^3 kg/m^3, i.e. g/cm^3.

The results can also be expressed as an intrinsic viscosity $[\eta]$, for example in order to compare copolymers of different average molecular masses for which it has been necessary to use different concentrations (see 6.2).

The intrinsic viscosity shall be calculated from the values of inherent viscosity obtained for concentrations c_1, c_2, c_3 ..., in the approximate ratio 1:2:3 ..., by a graphical method consisting of plotting the intrinsic-viscosity values (on the ordinate axis) against the concentration (on the abscissa) and extrapolating the curve to zero concentration. The intrinsic viscosity is read off the ordinate axis.

NOTE — It is recommended that the method of least squares be used for analysing the experimental values.

The intrinsic viscosity $[\eta]$ can also be calculated from one value of the reduced viscosity, for instance by the following equation:

$$[\eta]=\frac{I}{1+k'cI} \qquad \text{...(13)}$$

where k' is an experimental constant, usually between 0,2 and 0,3, which must be determined previously by measuring the reduced viscosity at two different concentrations and applying the equation:

$$k'=\frac{I_2-I_1}{I_1 I_2 (c_2-c_1)} \qquad \text{...(14)}$$

In the calculation of k', it may be preferable to use several concentrations and plot I against c, drawing a straight line through the points and selecting two positions on this line to give values for substitution in the equation for k'.

9.2 *K*-value

The *K*-value can be calculated from the relationship

$$K\text{-value} = 1\,000\,k \qquad \ldots(15)$$

where k is defined by the equations given in 3.3.6.

10 Test report

The test report shall contain the following particulars:

a) a reference to this part of ISO 1628 and the particular standard for the polymer tested, if one exists;

b) all details necessary for identification of the material tested, including type, source and manufacturer's code number;

c) the concentration of material in the solution used, a description of the solvent and details of the preparation of the solution;

d) details of the viscometer used;

e) the temperature of the test;

f) the test results;

g) the date of the test.

Annex A
(normative)

Cleaning of apparatus

It is essential that all apparatus coming into contract with the liquid under test is scrupulously clean. Any impurities such as dust, traces of liquid or filaments in the viscometer will cause false results to be obtained

Before the test, clean the viscometer and all apparatus used (glass vessels, pipettes, sintered-glass filters, rubber tubing, etc.) A suitable detergent may be adequate. Aqua regia (3 volumes concentrated hydrochloric acid and 1 volume concentrated nitric acid) may be required to remove inorganic residues from the glassware, and a suitable solvent may be required to remove oil and grease. Following this, dry the glass apparatus and then clean it with specially prepared chromic acid solution. Leave this solution in contact with the glassware overnight at a minimum temperature of 20 °C. For more active cleaning, warm the chromic acid gently on a water bath.

Remove the chromic acid and rinse at least five times with distilled or deionized water. Dry in an electric oven at a temperature no higher than 100 °C or rinse at least five times with acetone that has been distilled, dried and filtered, drying the apparatus with a slow stream of filtered dry air or, preferably, with a vacuum line.

Between successive determinations of the efflux times of samples of a similar nature, the viscometer may be cleaned by draining and thoroughly rinsing with a suitable volatile solvent that has been distilled and filtered. Dry the apparatus with a slow stream of filtered dry air, with a vacuum line, or in an electric oven at a temperature no higher than 100 °C. The effectiveness of the cleaning can be checked by establishing that the efflux times with a given solvent and viscometer remain constant.

If the next solution to be measured is of a polymer of the same type and of similar viscosity, it is permissible to drain the viscometer, wash it with the next solution to be measured and then fill it with a further quantity of this solution.

It is recommended that viscometers used for silicone fluids and fluorocarbons be reserved exclusively for use with these fluids and that these viscometers be subject to calibration checks at frequent intervals. Great care shall be taken to ensure that these fluids do not contaminate other apparatus, either directly or indirectly.

WARNING — Particular care should be taken when using aqua regia and chromic acid. Chromic acid is toxic. Appropriate action should also be taken to ensure the safe disposal of these acids. Goggles and protective gloves should be worn. Any splashes should be washed off the skin immediately with plenty of cold water. Inhalation of the vapours should be avoided.

Annex B
(normative)

Notes on sources of error

B.1 General

Guidance is given in this part of ISO 1628 on the determination of the viscometric properties of polymers in dilute solution as defined in clause 3. The measurement of these properties can usually be carried out with better reproducibility than that attained in absolute measurements of viscosity since many of the instrumental errors are approximately the same for both solvent and solution, and are effectively cancelled.

B.2 Principle of measurements

The relative viscosity and the other functions defined in clause 3 are calculated from the efflux times of solution and solvent. The liquid flows, under the force of gravity, through the capillary of a suspended-level Ubbelohde viscometer. The efflux times are taken to be proportional to the viscosity/density ratio of the fluids (see clause 4). This is equivalent to stating that the kinetic energy and other errors are negligible and that the density of the solvent differs negligibly from the density of the solutions.

B.3 Sources of error

The most important sources of error in capillary viscometry are related to

a) surface tension;

b) capillary end effects;

c) drainage effects;

d) viscous heat effects;

e) deviation of the viscometer from the vertical;

f) hydrostatic head variations;

g) losses by evaporation;

h) errors in the measurement of the concentration and efflux time;

i) shear effects;

j) errors in temperature stability and measurement;

k) kinetic energy.

Fortunately, most of these effects can be considered negligible for the proposed procedures. Errors due to surface tension, end effects and drainage effects are quite small in tests with organic solvents in the Ubbelohde viscometer, when relative measurements of viscosity are concerned[3]. Viscous heat effects are negligible when the capillary viscometer is operated by gravity. Errors due to deviation from the vertical and variations in the hydrostatic head are usually very small with this type of viscometer.

Solvent losses by evaporation and concentration errors depend on the particular test procedure and have to be taken into account.

The solvent efflux time should preferably be at least 70 s in order to measure it reproducibly, but it should not be longer than about 200 s in order to avoid long efflux times for the corresponding solutions.

The effect of shear rate can be serious, since polymer solutions are often non-Newtonian [4]. Strictly speaking, the measurements should be carried out at very low shear rates, but in the usual procedures the shear rate is as high as 1 000 s^{-1} or more. Fortunately, it can be shown [5] that shear rate effects are very small unless the molar mass of the polymer is higher than 10^6 g/mol, which seldom happens with commercial polymers. Furthermore, it would be impractical to take account of shear rate effects for the purposes of international standardization, and a better approach is to define the tolerance of the viscometer dimensions in such a way that the shear rate is closely reproducible in different laboratories testing the same polymer sample.

It may be concluded that the most important source of error in the measurements is the kinetic-energy term. Its value can be considerable. For example, it is approximately 15 % for chloroform in a standard Ubbelohde viscometer having a capillary diameter of 0,58 mm. The resultant error in the reduced viscosity can be of the order of 30 % and, for special work outside the scope of this part of ISO 1628, it might be appropriate to include a kinetic-energy correction.

B.4 Choice of viscometers

Since it is impractical to introduce a kinetic-energy correction into international standardization, it is necessary to choose the viscometer in such a way that

a) either the kinetic-energy term is negligible

b) or it is constant and reproducible in different laboratories.

It can be shown that the second condition cannot be realized easily. In fact, the maximum fractional hydrostatic pressure X spent in kinetic energy is given approximately by the equation

$$X = \frac{R^4 h g}{64 L^2 v^2} \qquad \text{...(16)}$$

where

R is the radius of the capillary;

h is the hydrostatic head;

g is the acceleration due to gravity;

L is the length of the capillary;

v is the viscosity/density ratio.

It is important to note in equation (16) that the efflux time and the reservoir volume by themselves do not influence the value of X.

For standard glass capillaries with diameters between 0,3 mm and 1,0 mm, the tolerance limits for the diameter[6] are usually ± 0,02 mm, corresponding to approximately ± 30 % and ± 8 %, respectively, of the value of X, whereas the mean hydrostatic head h is 120 mm ± 10 mm and the capillary length L is 90 mm ± 9 mm, corresponding to approximately ± 30 % of the value of X. The conclusion is that, unless the normal tolerances of standard capillaries are severely restricted, the kinetic-energy term cannot be made reproducible. It is therefore necessary to choose viscometers which minimize it.

In this part of ISO 1628, the maximum permitted value of X is fixed at 0,03 (approximately 4 % to 6 % of the reduced viscosity).

From equation (16), it can be seen that, at fixed values of h and L, the critical parameter which defines the magnitude of X is the ratio R^2/v.

By inserting

$h = 120$ mm,

$L = 90$ mm

and

$X < 0{,}03$

in equation (16), a relationship between the maximum capillary radius to be used and the value of v is obtained:

$$R^2 < 0{,}111\ v \qquad \text{...(17)}$$

or, for the diameter,

$$D^2 < 0{,}444\ v \qquad \text{...(18)}$$

where R and D are in mm and v is in $\text{mm}^2\cdot\text{s}^{-1}$.

It will be noted that equation (18) is dependent on the particular values ascribed to the mean hydrostatic head and the capillary length. These will vary between various designs of viscometer and in particular are slightly different for the Ubbelohde and DIN Ubbelohde versions.

Equation (18) permits calculation of the maximum theoretical diameter that could be used for a solvent whose viscosity/density ratio v is known at the temperature of measurement. It follows that, in order to determine the properties of polymers described in this part of ISO 1628, the viscosity/density ratio for the solvent chosen must be known. Table B.1 gives values at 25 °C for several of the established solvents for polymers, together with the calculated theoretical maximum capillary diameter for the Ubbelohde viscometer.

Equation (18) has also been used to select the recommended sizes of Ubbelohde and DIN Ubbelohde viscometers given in table 1. Other appropriate series of viscometers and sizes can be identified by substituting the relevant values into equation (16).

Table B.1 — Examples of viscosity/density ratios for established polymer solvents, and the corresponding maximum theoretical capillary diameters

Solvent	Viscosity/density ratio at 25 °C $mm^2 \cdot s^{-1}$	Maximum theoretical capillary diameter from equation (18) mm
Cyclohexanone	2,10	0,97
Dichloromethane	0,33	0,38
m-Cresol (meta-cresol)	13,10	2,41
Trichloromethane	0,36	0,40

Annex C
(informative)

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